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Figure 2J

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Supporting Information for Szyperski *et al.* (2002) *Proc. Natl. Acad. Sci. USA* **99** (12), 8009–8014. (10.1073/pnas.122224599).

Supporting Figure 11

Fig. 11. Experimental scheme for the 2D HBCB(CGCD)HD experiment. Rectangular 90° and 180° pulses are indicated by thin and thick vertical bars, respectively, and phases are indicated above the pulses. Where no rf phase is marked, the pulse is applied along x. The scaling factor k for ^{1}H chemical shift evolution during t_1 is set to 1.0. The high power 90° pulse lengths were: 5.8 ms for ^{1}H and 15.4 ms for ^{13}C. The first 180° pulse on ^{13}C prior to $t_1(^{13}$C) is applied at high power. Subsequently, the 90° pulse lengths of $^{13}C^b$ is adjusted to 66 ms. The 180° $^{13}C^b$ and $^{13}C^{aro}$ pulses are of gaussian-3 shape and 375 ms duration. WALTZ16 is used for decoupling of ^{1}H (rf field strength = 4.5 kHz) during the magnetization transfer from $^{13}C^a$ to $^{13}C^{aro}$, and the composite pulse decoupling scheme GARP is employed to decouple $^{13}C^{aro}$ (rf = 2.5 kHz) during acquisition. The ^{1}H rf carrier is placed at 0 ppm before the start of the semiconstant-time ^{1}H evolution period, and then switched to the water line at 4.78 ppm after the second 90° ^{1}H pulse. The ^{13}C rf carrier is set to 38 ppm during $w_1(^{13}C^b)$ and then switched to 131 ppm before the first 90° pulse on $^{13}C^{aro}$ (pulse labeled with f_4). The duration and strengths of the pulsed z-field gradients (PFGs) are: G1 (500 ms, 2 G/cm); G2 (1 ms, 22 G/cm); G3 (2 ms, 10 G/cm); G4 (1 ms, 5 G/cm); G5 (500 ms, 4 G/cm); G6 (1 ms, -14 G/cm); G7 (500 ms, -2G/cm). All PFG pulses are of rectangular shape. A recovery delay of at least 100 ms duration is inserted between a PFG pulse and an rf pulse. The delays are: t_1 = 1.8 ms, t_2 = 8.8 ms, t_3 = 71 ms, t_4 = 5.4 ms, t_5 = 4.2 ms, t_6 = 710 ms, t_7 = 2.5 ms. ^{1}H-frequency labeling is achieved in a semiconstant-time fashion with $t_1{}^a$ (0) = 1.7 ms, $t_1{}^b$ (0) = 1 ms, $t_1{}^c$ (0) = 1.701 ms, $Dt_1{}^a$ = 33.3 ms, $Dt_1{}^b$ = 19.3 ms, $Dt_1{}^c$ = -14 ms. Hence, the fractional increase of the semiconstant time period with t_1 equals to l = 1 + $Dt_1{}^c$ / $Dt_1{}^a$ = 0.58. Phase cycling: f_1 = x; f_2 = x; f_3 = x, y, -x, -y; f_4 = 4(x), 4(-x); f_5 (receiver) = x, -x, x, -x, -x, x, -x, x. Quadrature detection in $t_1(^{13}$C) is accomplished by altering the phases f_2 respectively, according to States-TPPI. For acquisition of central peaks derived from ^{13}C steady state magnetization, a second data set with f_1 = -x is collected. The sum and the difference of the two resulting data sets generate subspectra II and I, respectively, containing the central peaks and peak pairs.

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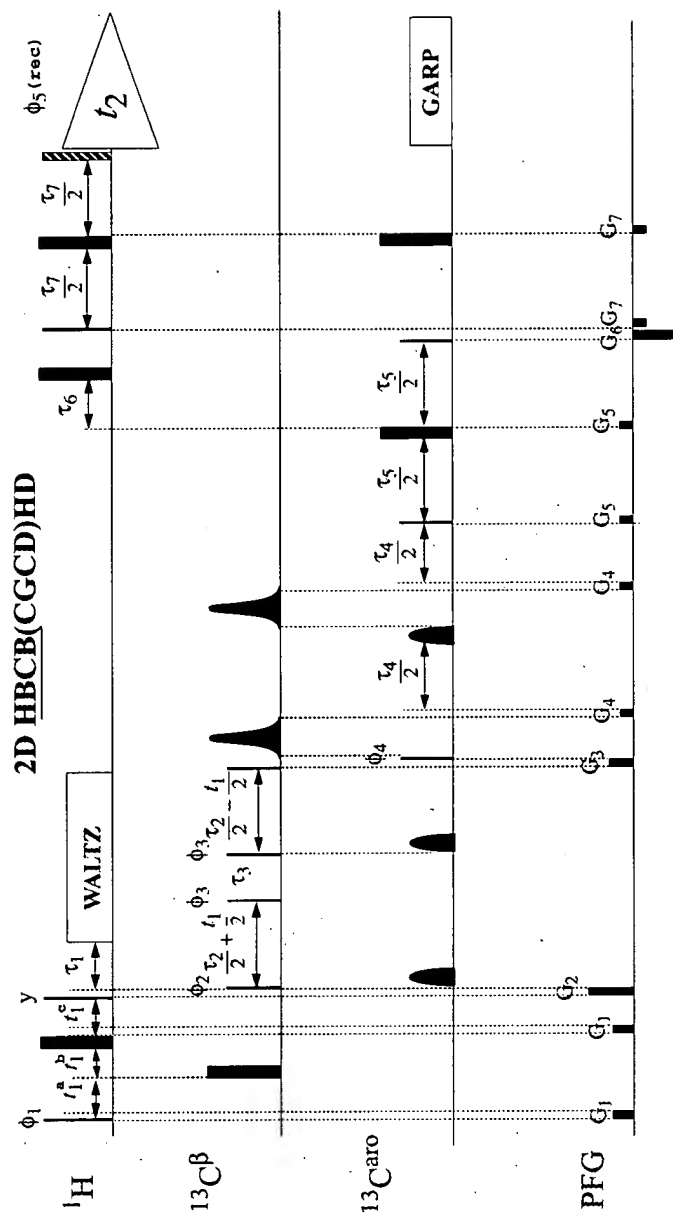
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2D HBCB(CGCD)HD

Figure 11